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3791

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

Analytical Chemistry Section
Building 306, BARC-East
Beltsville, Maryland 20705

SEP 14 1995

OFFICE OF
PREVENTION, PESTICIDES AND
TOXIC SUBSTANCES

MEMORANDUM

SUBJECT: Glyphosate Trimesium in or on Corn, Soybeans, Citrus Fruit, Stone Fruit and the Nut Crop Group (except Almonds). (MRID#s: 432736-04 and -05. Barcode D216509. Chemical No 128501. CBTS# 15715.)

FROM: Douglas Swineford, Chemist
Analytical Chemistry Section

THRU: Harvey K. Hundley, Head
Analytical Chemistry Section

THRU: Donald A. Marlow, Chief
Analytical Chemistry Branch
Biological and Economic Analysis Division (7503W)

TO: F. D. Griffith, Acting Section Head
Tolerance Petition Section III
Chemistry Branch I, Tolerance Support
Health Effects Division (7509C)

Introduction

The Analytical Chemistry Laboratory (ACL) validated the method: "Determination of Residues of the Trimethylsulfonium Cation in Agricultural Crops by Gas Chromatography" (Study Number GLYP-93-AM-04/Document Number RR93-105B). The matrices validated were pecans, corn grain, corn fodder, soybean seed and oranges.

Method Summary

The samples were extracted with an aqueous phosphate buffer and filtered. The extract is treated with basic anion-exchange resin to neutralize acids followed by phenylisocyanate and barium hydroxide to remove amino acids. The samples were cleaned up with cation-exchange resin and dealkylated prior to quantitation by gas chromatography (GC) using a flame photometric detector (FPD).

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Comments

1. Two experienced chemists can process a set of six samples in 12 hours, excluding the optional thermal cleanup (sec. 3.5) that may be necessary for some matrices. GC analysis time is approximately 5 minutes per injection.

2. The limit of detection from visual inspection of the chromatograms is estimated to be 0.01 ppm for the matrices tested.

3. No special safety hazards were noticed. Normal laboratory safety procedures were followed.

4. Standards were obtained from the registrant (Zeneca Ag Products, Richmond, CA 94840-0023). The EPA standards repository in RTP, North Carolina indicated they have a small amount of the standard available.

5. ACL has received various analytical methods this year for the validation of the trimethylsulfonium cation from Zeneca Ag Products for multiple raw agricultural commodities. Has the registrant considered the feasibility of combining the various approaches into a single method?

Separate comments

6. If the above comments are taken into consideration and incorporated with additional comments noted in the laboratory pre-review, the method would meet 40 CFR 158 and EPA's requirements as published in the Pesticide Assessment Guidelines, Subdivision "O" for Residue Chemistry, Part 171-4(b) as an enforcement method.

7. Recovery data, example chromatograms, pre-review checklist and pre-review comments are attached.

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VALIDATION REPORT TRIMETHYLSULFONIUM CATION (TMS)

Commodity	Chemical Added	PPM Added	PPM Found	%Recovery
Corn Grain	Control	0	N.D.*	---
	Control	0	N.D.*	---
	TMS	0.05	0.0463	92.6
	TMS	0.05	0.0465	93.0
	TMS	0.10	0.0854	85.4
	TMS	0.10	0.0820	82.0

* N.D. = < 0.01 ppm

Commodity	Chemical Added	PPM Added	PPM Found	%Recovery
Corn Fodder	Control	0	N.D.*	---
	Control	0	N.D.*	---
	TMS	0.10	0.1030	103
	TMS	0.10	0.1021	102
	TMS	1.0	1.0119	101
	TMS	1.0	0.8960	89.6
	TMS	2.0	1.9367	96.8
	TMS	2.0	1.8282	91.4

N.D.* = < 0.01 ppm

VALIDATION REPORT TRIMETHYLSULFONIUM CATION (TMS) (continued)

Commodity	Chemical Added	PPM Added	PPM Found	%Recovery
Soybean Seed	Control	0	N.D.*	---
	Control	0	N.D.*	---
	TMS	0.05	0.0426	85.2
	TMS	0.05	0.0478	95.6
	TMS	0.50	0.4017	80.3
	TMS	0.50	0.4447	88.9
	TMS	1.0	0.9494	94.9
	TMS	1.0	0.9743	97.4

* N.D. = < 0.01 ppm

Commodity	Chemical Added	PPM Added	PPM Found	%Recovery
Oranges	Control	0	N.D.*	---
	Control	0	N.D.*	---
	TMS	0.05	0.0450	90.0
	TMS	0.05	0.0437	87.4
	TMS	0.50	0.5290	106
	TMS	0.50	0.5050	101
	TMS	1.0	1.1752	118
	TMS	1.0	1.1115	111

* N.D. = < 0.01 ppm

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VALIDATION REPORT TRIMETHYLSULFONIUM CATION (TMS) (continued)

Commodity	Chemical Added	PPM Added	PPM Found	%Recovery
Pecans	Control	0	N.D.*	---
	Control	0	N.D.*	---
	TMS	0.05	0.0510	102
	TMS	0.05	0.0544	109
	TMS	0.10	0.1070	107
	TMS	0.10	0.1026	103

* N.D. = < 0.01 ppm

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Modifications to method:

None.

Special precautions to be taken:

Handle all chemicals in a safe manner.

Source of analytical standard:

Zeneca Ag Products, Richmond, CA 94804-0023.

If derivatized standard, give source:

N/A

Instrument for quantitation:

GC/FPD

Instrument for confirmation:

N/A

If instrument parameters differ form those given in method, list parameters used:

N/A

Commercial sources of any special chemicals of apparatus:

N/A

Additional comments:

See report.

Chromatograms:

Copies attached.

TMV Pre-Review of Glyphosate- Trimesium

Reviewed By: Dallas Wright, Jr. DPW

Date: June 15, 1995

Laboratory Assignment Number: B95-34,36,37,38,39

Analyte: Trimethylsulfonium Cation

Commodities: Pecans, Corn grain, ~~Soybean Hay~~ Corn Fodder, Soybean Seed, and Oranges

Petitioner: Zeneca Ag Products

Method: "Determination of Residues of the Trimethylsulfonium Cation in Agricultural Crops by Gas Chromatography;" Dec. 30, 1993; RR 93-105B

Section 3: Analytical Procedure

3.2.2: In the note for this step there are suggestions for filtering the extract; however, there is no filtration step in the extraction for oily crops.

3.3.5: Is it necessary to continue the evacuation of the flask for 1-2 minutes as in step 3.3.4? Should the filter cake be removed before the second filtration? (1) Ask ACS what they did + ask registrant then circulate

3.5 Thermal Cleanup: Normally ACL safety regulations do not allow overnight heating or extractions. However, this step should be included in the validation. Is it possible to reduce the time to 8 or 9 hours? (1)

3.6 Dealkylation: 1) Since the toluene is added before the sample is heated, it is possible that the volume will change due to leaking during the heating. Since there is only 0.5 to 1 ml of toluene to start with, a small loss during heating could result in a large percentage change to the final volume used for calculations.

Section 5: Calculations: The instructions are misleading and seem to indicate that it would be appropriate to use the average response factor when using an FPD. The average response factor method for calculating cannot be used when the samples are quantitated on an FPD in sulfur mode, since the response is not linear. The method should clearly state that the analyst must use either a linear regression based on the square root of the response or closely match standard and sample responses.

Section 8.4 Lower Limit of Quantitation: 1) The LOQ for this method is 0.05 ppm for food crops and 0.1 ppm for feed items.

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Section 9 Conclusions: The method has not been tested for samples less than the LOQ and greater than 10 times the LOQ. Some of the requested spiking levels are greater than 10 times the LOQ. The following table summarizes the data for each commodity requested, the LOQ, and the requested spiking levels:

Commodity	LOQ (PPM)	10x LOQ (PPM)	Data Provided (PPM)	Requested Levels (PPM)
Pecans	0.05	0.5	0.05; 0.1	0.05; 0.10
Corn Grain	0.05	0.5	0.05; 0.5	0.05; 0.10
Corn Fodder	0.1	1.0	0.1; 1.0	0.1; 1.0; 2.0
Soybean Seed	0.1	1.0	0.05; 0.1	0.05; 0.5; 1.0
Oranges	0.05	0.5	0.05; 0.09; 0.23	0.05; 0.5; 1.0

Recovery Data: Not all recovery data was within the range of 70-120%. The following list summarizes the data: Pecans: 62-112%; Corn grain: 81-118%; Corn Fodder: 85-115%; Soybean seed: 74-110%; Oranges: 104-125%

ILV Data: The Independent Laboratory Validation was run by Morse Laboratories, Inc. on corn grain (0.05 and 0.5 ppm), forage (0.1, and 1.0 ppm), and fodder (0.1, and 1.0 ppm). Acceptable recoveries were obtained on the first set of samples run.

ANALYTICAL CHEMISTRY BRANCH
SCREEN FOR RESIDUE METHODS FOR TMV

1. LABORATORY ASSIGNMENT NUMBER: B95-34, 36-39

2. PP#: 9F03796, 0F03860, 3F04238, 4F04343

3. TECHNICAL REVIEWER: Dallas Wright, Jr.

4. DATE: June 19, 1995

5. ANALYTES/LEVEL: See Review

6. COMMODITIES: Pecans, Corn Grain, Corn Fodder, Soybean Seed, Oranges

7. METHOD: Determination of Residues of the Trimethylsulfonium Cation in Agricultural crops by Gas Chromatography

The Analytical Chemistry Section has been asked to screen the residue chemistry methods submitted by the registrant in order to determine if they contain the essential requirements identified in the Residue Chemistry Guidelines. Full scientific review and laboratory evaluation of those methods will take place after the initial screen. The following items need to be resolved before the analytical method can be evaluated.

	YES	NO
1. Does the method use exotic equipment and/or supplies that are not commercially available in the U.S.?		✓
2. Does the method require any new equipment before the laboratory work begins?		✓
3. Are chromatograms included?		
a. Is (are) peak(s) of interest sufficiently resolved from other peaks?	✓	
b. Has registrant included chromatograms of analyses at or below tolerance on all crop types for which tolerance is requested by HED? See Review	✓	✓
c. Do the control samples have reasonably low levels of the analyte in relation to the proposed tolerance?	✓	
d. Is the method sufficiently sensitive and specific to measure and identify the residues at levels specified by HED in the TMV request?	✓	

	YES	NO
4. Has recovery data been provided to ACL for the residues that are specified in the TMV request? See Review		✓
5. Are recovery values between 70% and 120% at all levels and for all commodity types? See Review		✓
6. Are all procedures clearly written with no ambiguities so that the method can be run without communication with the registrant? See Review		✓
7. Does the method require correction for a sample of the untreated commodities or a blank?		✓
8. Does the method require the use of an internal or procedural standard to compensate for lost analyte during analysis?		✓
9. Are 2nd laboratory validation data provided with the method?	✓	
10. Are there any deficiencies other than those covered above that would prevent ACS from conducting a method trial?		✓
11. Is this method suitable for validation testing?	✓	

Any deficiencies/problems noted for any above items should be addressed in the full scientific review of this method to be attached as an addendum.

[signature] 6/19/95

Signature Date

The following is to be completed by the analyst performing the TMV.

12. Can a set of 6 samples be run within 24 hours?	✓	
13. a. Are standards available at RTP repository?	✓	
b. Are derivatized analytical reference standards available?	N/A	

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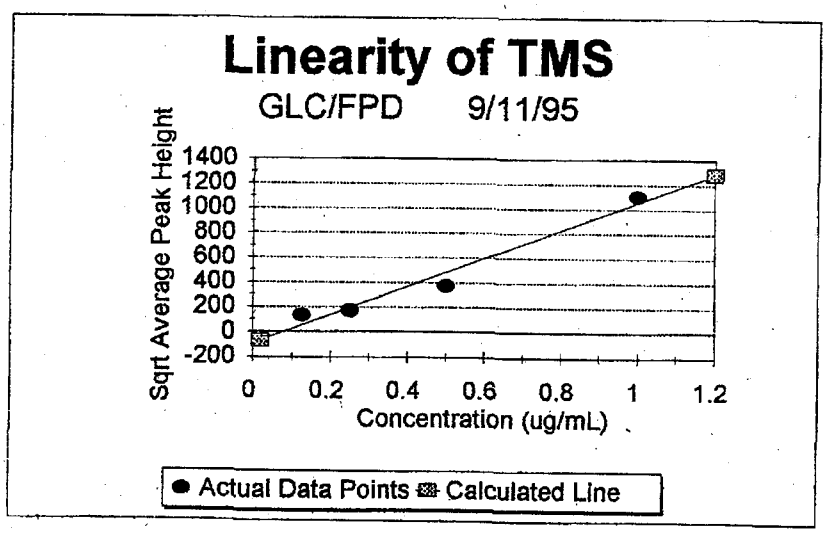
B95-34-35

Linearity of TMS
GLC/FPD
9/11/95

ug/mL	PK. HT. 1	PK. HT.2	AVE PK. HT.	Sqrt Avg Pk Ht	Sqrt CALC. PK. HT.
0.1250	18082.0	17314.0	17698.0	133.0338	
0.2500	29935.0	29323.0	29629.0	172.1308	
0.5000	128467.0	146861.0	137664.0	371.0310	
1.0000	1223028.0	1223179.0	1223103.5	1105.9401	
0.0200					-69.6693
1.2000					1285.0712

Regression Output:

Constant		-92.6310293
Std Err of Y Est		104.8272386
R Squared		0.964211424
No. of Observations		4
Degrees of Freedom		2
X Coefficient(s)	1148.08523	
Std Err of Coef.	156.4030464	



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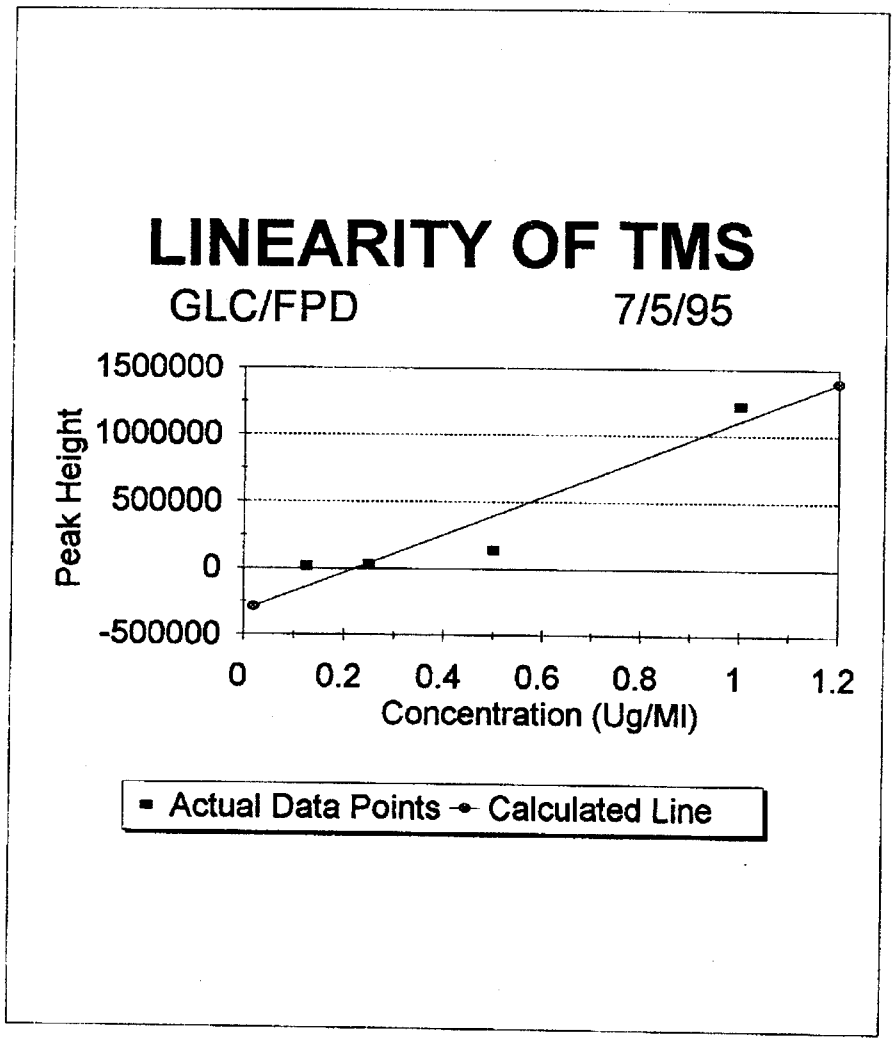
Linearity of TMS
GLC - FPD
7/5/95

ug/mL	PK. HT 1	PK. HT 2	AVG PK. HT	CALC. PK. HT
0.125	18082.0	17314.0	17698.0	
0.250	29935.0	29323.0	29629.0	
0.500	128467.0	146861.0	137664.0	
1.000	1223028.0	1223179.0	1223103.5	
0.020				-288817.68
1.200				1396291.48

Regression Output:		
Constant		-317378.847826
Std Err of Y Est		228393.556228
R Squared		0.89776271334
No. of Observations		4
Degrees of Freedom		2
Std Err of Coef.	0.089136462	
X Coefficient(s)	1428058.609	
Std Err of Coef.	340764.943	



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```
RUN #  545      AUG  5, 1995  13:32:16
START
                    IF
                    IF
                    IF                  1.163
     TIMETABLE STOP
```

```
RUN#   545          AUG  5, 1995  13:32:16

SAMPLE NAME: DMSS STD          SAMPLE#    1
0.25 UG/ML


B95/34 THRU 39 (SOYBEAN SEED) 2UL INJ

HEIGHT%
       RT     HEIGHT TYPE  WIDTH    HEIGHT%
TMS  1.163     49240   BV   .015  100.00000

TOTAL HEIGHT=  49240
MUL FACTOR=1.0000E+00



RUN PARAMETERS
ZERO    =  20
ATT 2^  =  4
CHT SP  =  1.0
AR REJ  =  6000
THRSH   =  3
PK WD   =  0.02
```

```
RUN #  546      AUG  5, 1995  13:35:56
START
                    IF
                    IF
                    IF  1.215
     TIMETABLE STOP
```

```
RUN#   546          AUG  5, 1995  13:35:56

SAMPLE NAME: CONTROL #1        SAMPLE#    2
FINAL VOL 1ML/5G ALIQUOT


B95/34 THRU 39 (SOYBEAN SEED) 2UL INJ

NO RUN PEAKS STORED


RUN PARAMETERS
```

13

```
RUN PARAMETERS
ZERO    =  20
ATT 2^  =  4
CHT SP  =  1.0
AR REJ  =  6000
THRSH   =  3
PK WD   =  0.02

RUN #  546      AUG  5, 1995  13:35:56
START
                        IF
                        IF
                        IF 1.215
     TIMETABLE STOP

RUN#   546            AUG  5, 1995  13:35:56

SAMPLE NAME: CONTROL #1        SAMPLE#    2
FINAL VOL 1ML/5G ALIQUOT

B95/34 THRU 39 (SOYBEAN SEED) 2UL INJ

NO RUN PEAKS STORED


RUN PARAMETERS
ZERO    =  20
ATT 2^  =  4
CHT SP  =  1.0
AR REJ  =  6000
THRSH   =  3
PK WD   =  0.02

RUN #  647      AUG  5, 1995  13:39:36
START
                        IF
                        IF
                        IF 1.052
     TIMETABLE STOP

RUN#   547            AUG  5, 1995  13:39:36

SAMPLE NAME: CONTROL #1        SAMPLE#    2
FINAL VOL 1ML/5G ALIQUOT

B95/34 THRU 39 (SOYBEAN SEED) 2UL INJ
```

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```
RUN PARAMETERS
ZERO    =  20
ATT 2^  =  4
CHT SP  =  1.0
AR REJ  =  6000
THRSH   =  3
PK WD   =  0.02


RUN #  554      AUG  5, 1995  14:05:18
START
                    IF
                    IF
                    IF                  1.155
     TIMETABLE STOP


RUN#   554          AUG  5, 1995  14:05:18

SAMPLE NAME: 0.05PPM SPK#      SAMPLE#    6
FINAL VOL 1ML/5G ALIQUOT


B95/34 THRU 39 (SOYBEAN SEED) 2UL INJ

HEIGHT%
      RT     HEIGHT TYPE  WIDTH    HEIGHT%
   1.155      40259   PV    .018  100.00000

TOTAL HEIGHT=  40259
MUL FACTOR=1.0000E+00



RUN PARAMETERS
ZERO    =  20
ATT 2^  =  4
CHT SP  =  1.0
AR REJ  =  6000
THRSH   =  3
PK WD   =  0.02


RUN #  555      AUG  5, 1995  14:09:01
START
                    IF
                    IF
                    IF                  1.155
     TIMETABLE STOP


RUN#   555          AUG  5, 1995  14:09:01

SAMPLE NAME: 0.05PPM SPK#      SAMPLE#    6
FINAL VOL 1ML/5G ALIQUOT
```

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1.154 27393 PU .017 100.00000

TOTAL HEIGHT= 27393
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 20
ATT 2^ = 4
CHT SP = 1.0
AR REJ = 6000
THRSH = 3
PK WD = 0.02

RUN # 560 AUG 5, 1995 14:27:22
START

IF
IF
1.152
IF

TIMETABLE STOP

RUN# 560 AUG 5, 1995 14:27:22

SAMPLE NAME: 0.5PPM SPK#2 SAMPLE# 9
FINAL VOL 10ML/5G ALIQUOT

B95/34 THRU 39 (SOYBEAN SEED) 2UL INJ

HEIGHT%

RT	HEIGHT	TYPE	WIDTH	HEIGHT%
1.152	34198	PU	.017	100.00000

TOTAL HEIGHT= 34198
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 20
ATT 2^ = 4
CHT SP = 1.0
AR REJ = 6000
THRSH = 3
PK WD = 0.02

RUN # 561 AUG 5, 1995 14:31:03
START

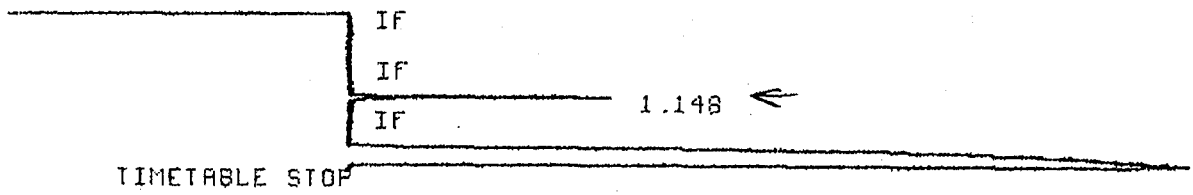
IF
IF
1.061
IF
1.152

16

TOTAL HEIGHT= 32333
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 20
ATT 2^ = 4
CHT SP = 1.0
AR REJ = 6000
THRSH = 3
PK WD = 0.02

RUN # 567 AUG 5, 1995 14:53:11
START



RUN# 567 AUG 5, 1995 14:53:11

SAMPLE NAME: 1PPM SPK#2 SAMPLE# 12
FINAL VOL 20ML/5G ALIQUOT

B95/34 THRU 39 <SOYBEAN SEED> 2UL INJ

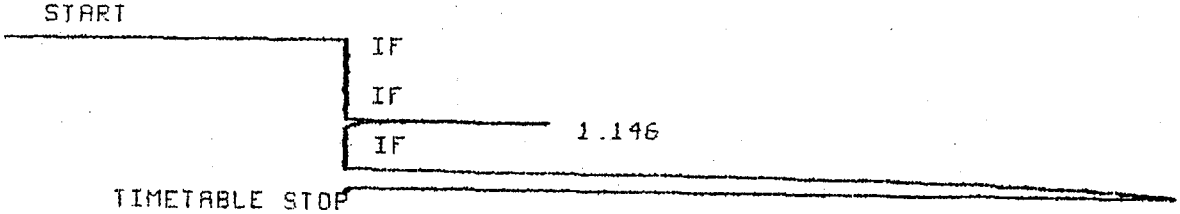
HEIGHT%

RT	HEIGHT	TYPE	WIDTH	HEIGHT%
1.148	32041	PV	.018	100.00000

TOTAL HEIGHT= 32041
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 20
ATT 2^ = 4
CHT SP = 1.0
AR REJ = 6000
THRSH = 3
PK WD = 0.02

RUN # 568 AUG 5, 1995 14:56:48
START



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SAMPLE NAME: DMS STD SAMPLE# 7
0.25 UG/ML

B95/34 THRU 39 (CORN FODDER) 2UL INJ

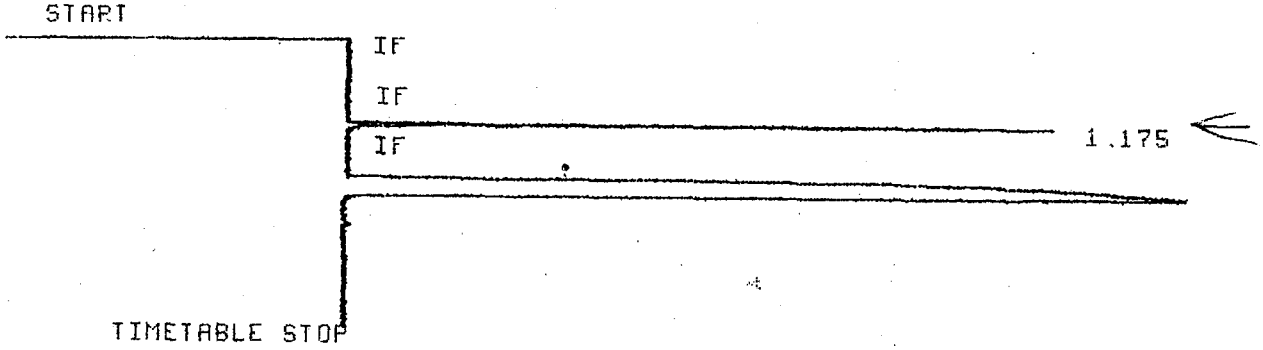
HEIGHT%

RT	HEIGHT	TYPE	WIDTH	HEIGHT%
1.174	95550	VV	.015	100.00000

TOTAL HEIGHT= 95550
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 20
ATT 2^ = 4
CHT SP = 1.0
AR REJ = 6000
THRSH = 3
PK WD = 0.02

RUN # 371 JUL 27, 1995 16:34:23
START



RUN# 371 JUL 27, 1995 16:34:23

SAMPLE NAME: DMS STD SAMPLE# 7
0.25 UG/ML 2ul inj

B95/34 THRU 39 (CORN FODDER) 2UL INJ

HEIGHT%

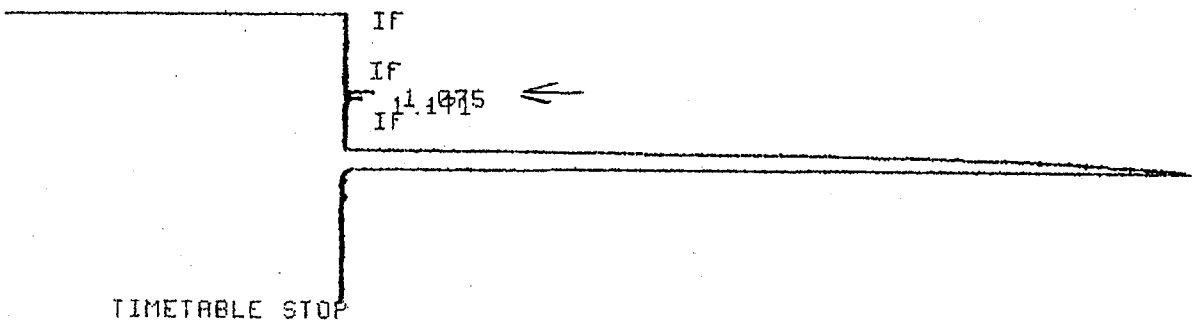
	RT	HEIGHT	TYPE	WIDTH	HEIGHT%
TMS	1.175	84906	PB	.014	100.00000

TOTAL HEIGHT= 84906
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 20
ATT 2^ = 4
CHT SP = 1.0

RUN PARAMETERS
ZERO = 20
ATT 2^ = 4
CHT SP = 1.0
AR REJ = 6000
THRSH = 3
PK WD = 0.02

RUN # 361 JUL 27, 1995 15:39:45
START



RUN# 361 JUL 27, 1995 15:39:45

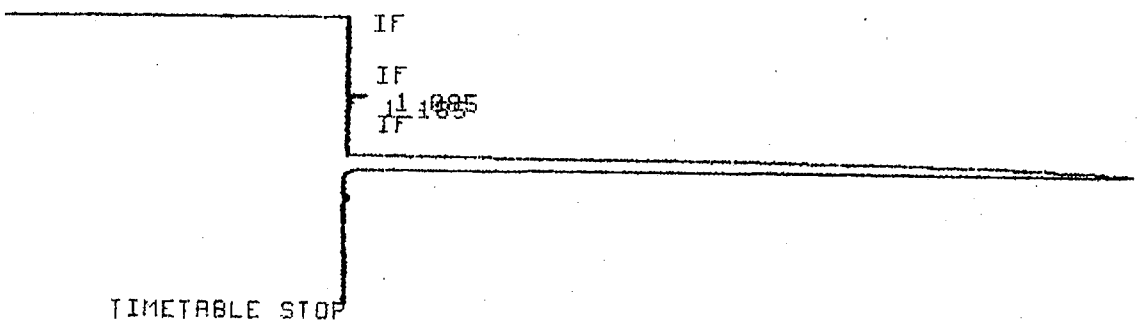
SAMPLE NAME: CONTROL #1 SAMPLE# 2
FINAL VOL 2ML/5G ALIQUOT

B95/34 THRU 39 (CORN FODDER) 2UL INJ

NO RUN PEAKS STORED

RUN PARAMETERS
ZERO = 20
ATT 2^ = 4
CHT SP = 1.0
AR REJ = 6000
THRSH = 3
PK WD = 0.02

RUN # 362 JUL 27, 1995 15:45:10
START

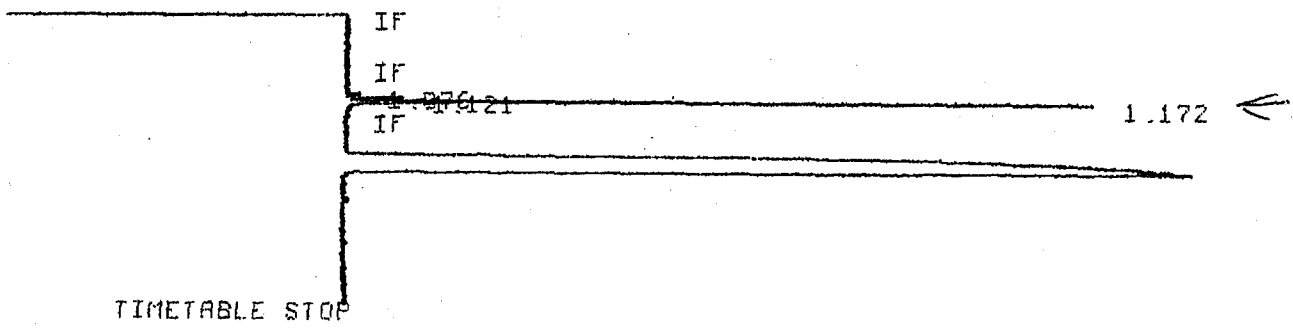


RUN# 362 JUL 27, 1995 15:45:10

SAMPLE NAME: CONTOL #2 SAMPLE# 3

```
RUN PARAMETERS
ZERO    =  20
ATT 2^  =  4
CHT SP  =  1.0
AR REJ  =  6000
THRSH   =  3
PK WD   =  0.02
```

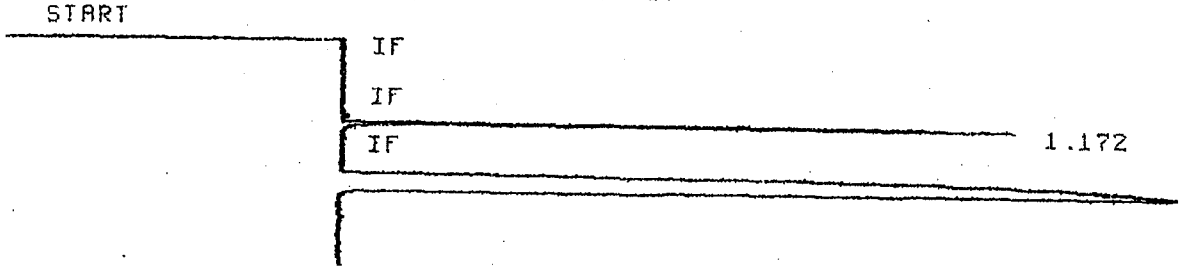
```
RUN #  367      JUL 27, 1995  16:12:25
START
```




```
RUN#   367            JUL 27, 1995  16:12:25

SAMPLE NAME: 0.1 SPK#1          SAMPLE#    5
FINAL VOL 2ML/5G ALIQUOT


B95/34 THRU 39 (CORN FODDER) 2UL INJ

HEIGHT%
      RT     HEIGHT TYPE  WIDTH    HEIGHT%
   1.121       6857   PU    .015    7.12342
   1.172      89403   VB    .014   92.87658

TOTAL HEIGHT=  96260
MUL FACTOR=1.0000E+00
```

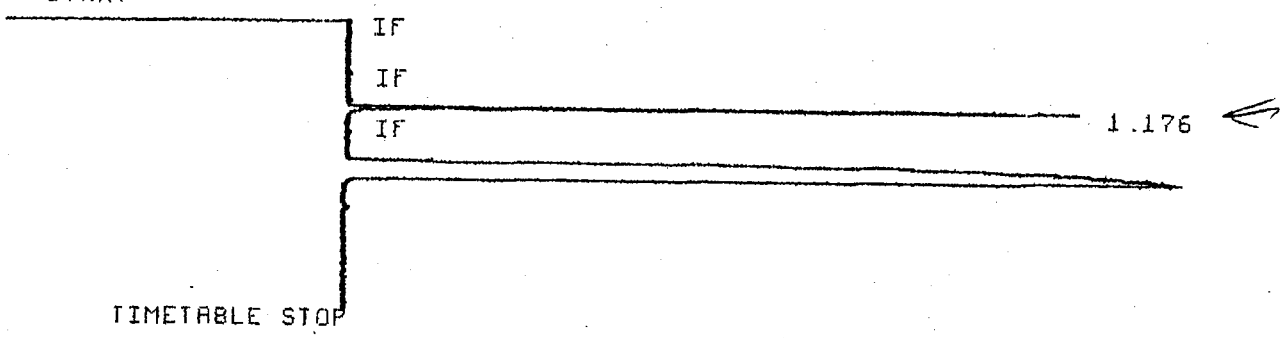
```
RUN PARAMETERS
ZERO    =  20
ATT 2^  =  4
CHT SP  =  1.0
AR REJ  =  6000
THRSH   =  3
PK WD   =  0.02
```

```
RUN #  368      JUL 27, 1995  16:17:57
START
```



ATT 2^ = 4
CHT SP = 1.0
AR REJ = 6000
THRSH = 3
PK WD = 0.02

RUN # 373 JUL 27, 1995 16:45:17
START



RUN# 373 JUL 27, 1995 16:45:17

SAMPLE NAME: 1PPM SPK#1 SAMPLE# 8
FINAL VOL 20ML/5G ALIQUOT

B95/34 THRU 39 (CORN FODDER) 2UL INJ

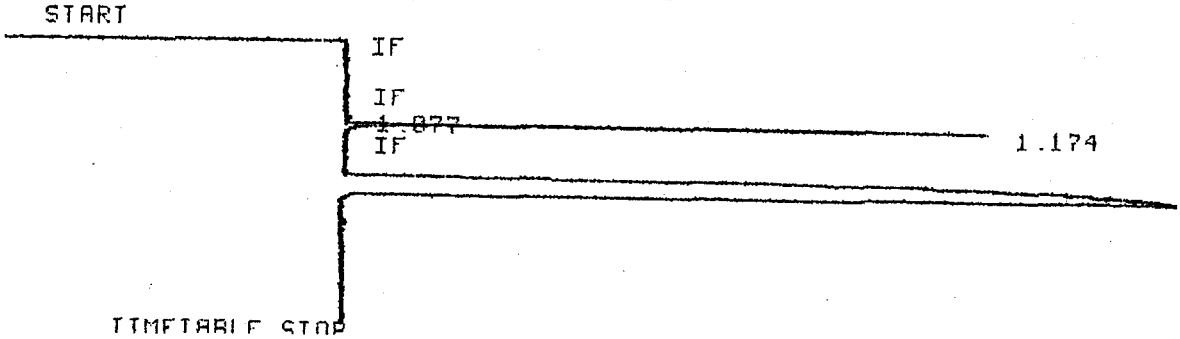
HEIGHT%

RT	HEIGHT	TYPE	WIDTH	HEIGHT%
1.176	87658	PB	.014	100.00000

TOTAL HEIGHT= 87658
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 20
ATT 2^ = 4
CHT SP = 1.0
AR REJ = 6000
THRSH = 3
PK WD = 0.02

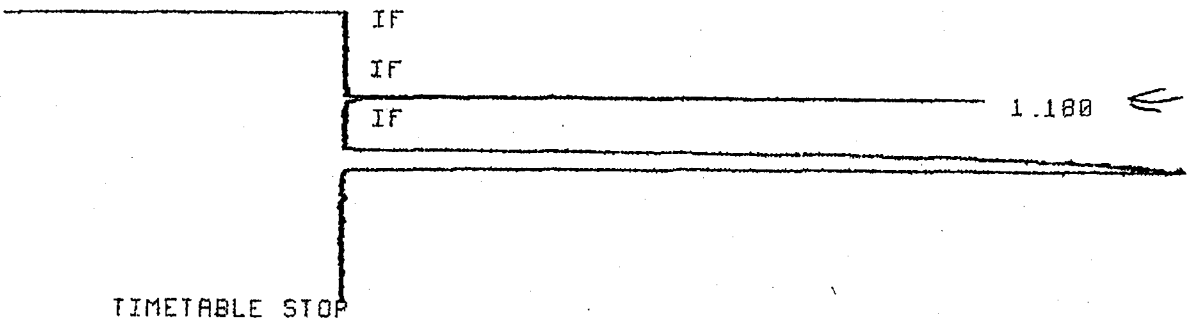
RUN # 374 JUL 27, 1995 16:50:45
START



21

RUN PARAMETERS
ZERO = 20
ATT 2^ = 4
CHT SP = 1.0
AR REJ = 6000
THRSH = 3
PK WD = 0.02

RUN # 380 JUL 27, 1995 17:23:36
START



RUN# 380 JUL 27, 1995 17:23:36

SAMPLE NAME: 2PPM SPK#2 SAMPLE# 12
FINAL VOL 40ML/5G ALIQUOT

B95/34 THRU 39 (CORN FODDER) 2UL INJ

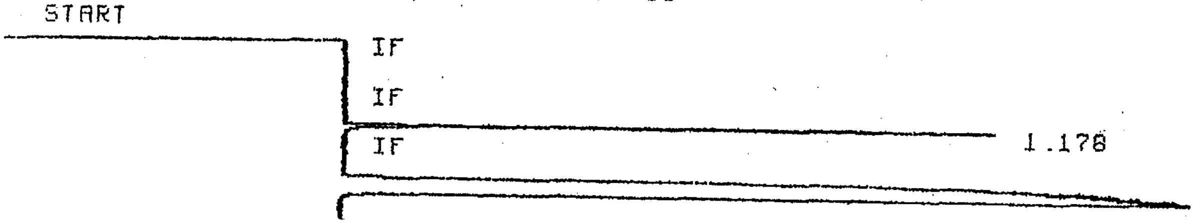
HEIGHT%

RT	HEIGHT	TYPE	WIDTH	HEIGHT%
1.180	76587	PB	.013	100.00000

TOTAL HEIGHT= 76587
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 20
ATT 2^ = 4
CHT SP = 1.0
AR REJ = 6000
THRSH = 3
PK WD = 0.02

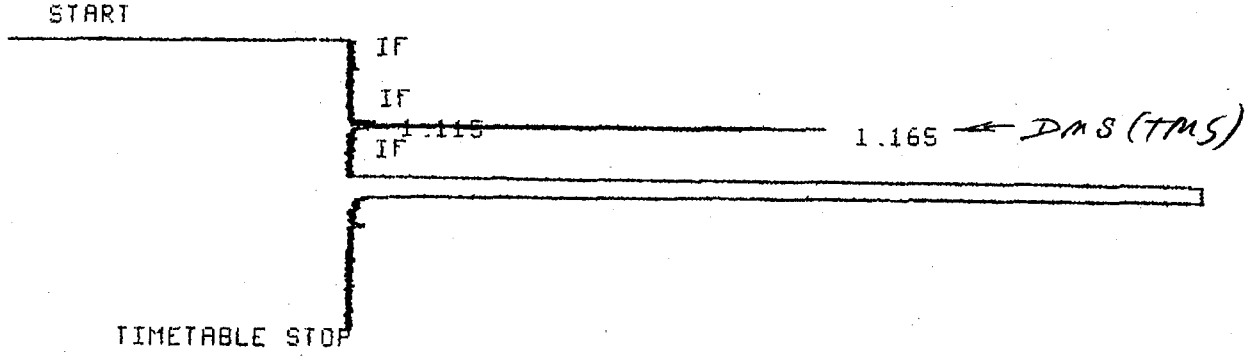
RUN # 381 JUL 27, 1995 17:29:03
START



22

RUN PARAMETERS
ZERO = 20
ATT 2^ = 3
CHT SP = 1.0
AR REJ = 6000
THRSH = 3
PK WD = 0.02

RUN # 147 JUL 12, 1995 09:24:13
START



RUN# 147 JUL 12, 1995 09:24:13

SAMPLE NAME: TMS STD SAMPLE# 1
0.25 UG/ML 2 ul inj

B95-34 THRU 39(CORN GRAIN)

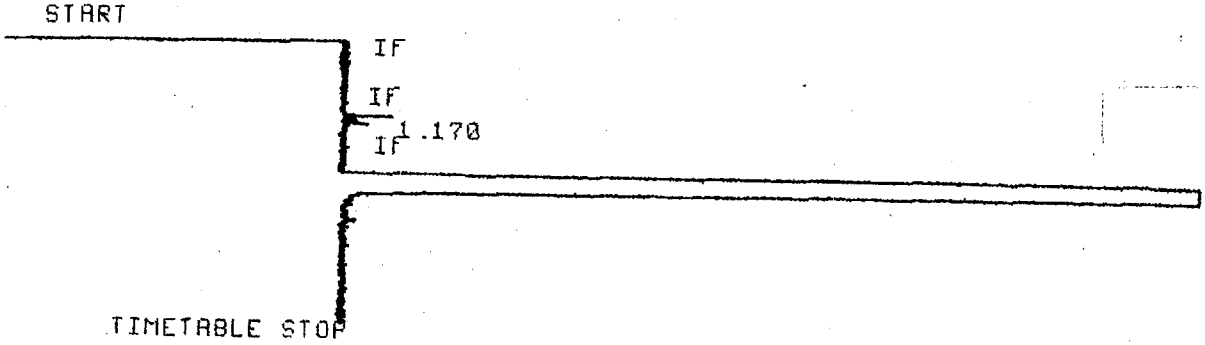
HEIGHT%

	RT	HEIGHT	TYPE	WIDTH	HEIGHT%
TMS	1.165	28482	PB	.015	100.00000

TOTAL HEIGHT= 28482
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 20
ATT 2^ = 3
CHT SP = 1.0
AR REJ = 6000
THRSH = 3
PK WD = 0.02

RUN # 148 JUL 12, 1995 09:29:43
START

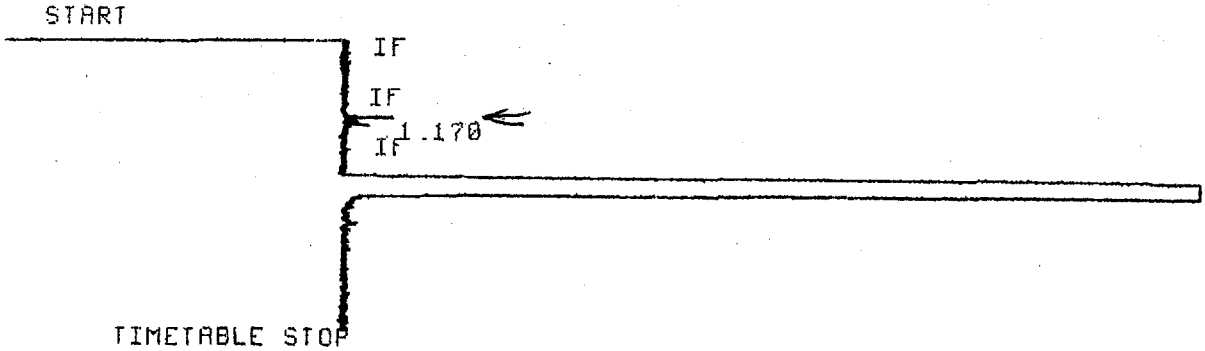


23

TOTAL HEIGHT= 28482
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 20
ATT 2^ = 3
CHT SP = 1.0
AR REJ = 6000
THRSH = 3
PK WD = 0.02

RUN # 148 JUL 12, 1995 09:29:43
START



RUN# 148 JUL 12, 1995 09:29:43

SAMPLE NAME: CONTROL#1 SAMPLE# 2
2UL INJ/FINAL VOL 1ML/5G ALIQUOT

B95-34 THRU 39(CORN GRAIN)

NO RUN PEAKS STORED

RUN PARAMETERS
ZERO = 20
ATT 2^ = 3
CHT SP = 1.0
AR REJ = 6000
THRSH = 3
PK WD = 0.02

RUN # 149 JUL 12, 1995 09:35:12
START

B95-34 THRU 39(CORN GRAIN)

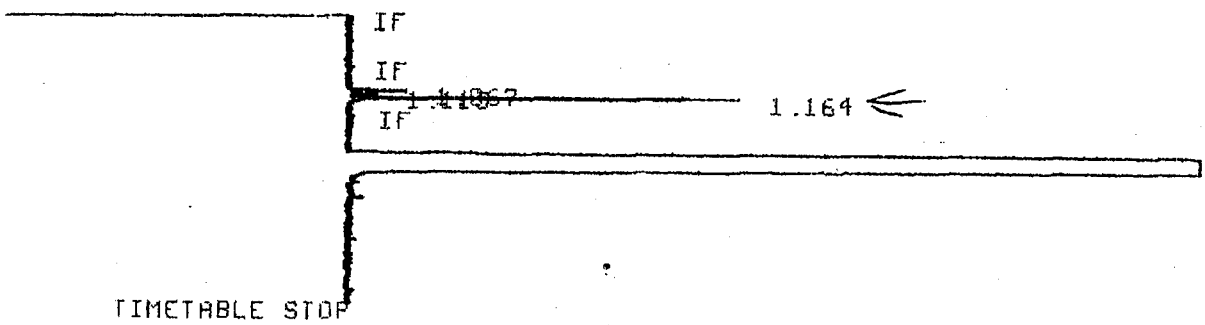
HEIGHT%

RT	HEIGHT	TYPE	WIDTH	HEIGHT%
1.164	25518	VV	.016	100.00000

TOTAL HEIGHT= 25518
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 20
ATT 2^ = 3
CHT SP = 1.0
AR REJ = 6000
THRSH = 3
PK WD = 0.02

RUN # 154 JUL 12, 1995 10:02:31
START



RUN# 154 JUL 12, 1995 10:02:31

SAMPLE NAME: 0.05PPM SPK1 SAMPLE# 5
2UL INJ/ FINAL VOL 1ML/ 5G ALIQUOT

B95-34 THRU 39(CORN GRAIN)

HEIGHT%

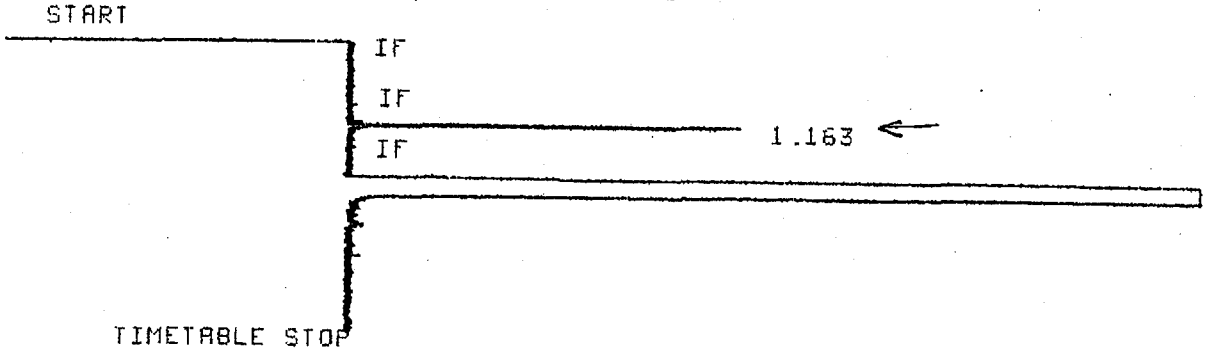
RT	HEIGHT	TYPE	WIDTH	HEIGHT%
1.164	23776	VV	.015	100.00000

TOTAL HEIGHT= 23776
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 20
ATT 2^ = 3
CHT SP = 1.0
AR REJ = 6000
THRSH = 3
PK WD = 0.02

RUN PARAMETERS
ZERO = 20
ATT 2^ = 3
CHT SP = 1.0
AR REJ = 6000
THRSH = 3
PK WD = 0.02

RUN # 161 JUL 12, 1995 10:41:03
START



RUN# 161 JUL 12, 1995 10:41:03

SAMPLE NAME: 0.1SPK1 SAMPLE# 8
2UL INJ/ FINAL VOL2ML/ 5G ALIQUOT

B95-34 THRU 39(CORN GRAIN)

HEIGHT%

RT	HEIGHT	TYPE	WIDTH	HEIGHT%
1.163	23813	PV	.016	100.00000

TOTAL HEIGHT= 23813
MUL FACTOR=1.0000E+00

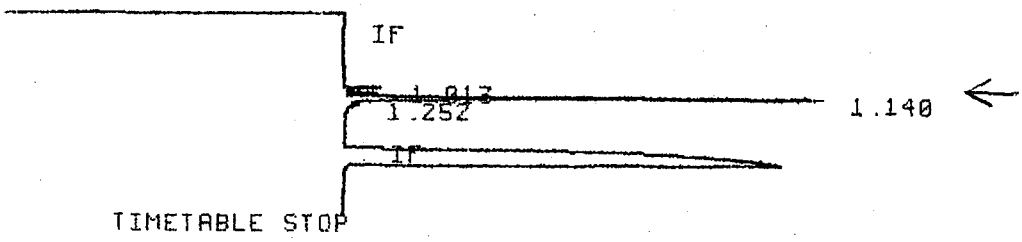
RUN PARAMETERS

26

TOTAL HEIGHT= 158790
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 20
ATT 2^ = 5
CHT SP = 1.0
AR REJ = 2000
THRSH = 3
PK WD = 0.06

RUN # 1217 AUG 29, 1995 10:54:38
START



Storing processed peaks to A:Q14398EF.PRO

RUN# 1217 AUG 29, 1995 10:54:38

SAMPLE NAME: 0.25UG/ML SAMPLE# 1
DMS STD

PEAK FILE : A:Q14398EF.PRO

B95/34-39 (PECANS) 2UL INJ

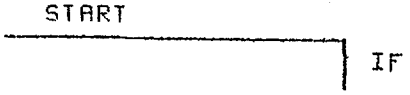
HEIGHT%

	RT	HEIGHT	TYPE	WIDTH	HEIGHT%
	1.013	8814	BB	.015	6.79301
tms	1.140	118403	BB	.022	91.25402
	1.252	2534	BB	.016	1.95297

TOTAL HEIGHT= 129751
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 20
ATT 2^ = 5
CHT SP = 1.0
AR REJ = 2000
THRSH = 3
PK WD = 0.06

RUN # 1218 AUG 29, 1995 10:59:49
START



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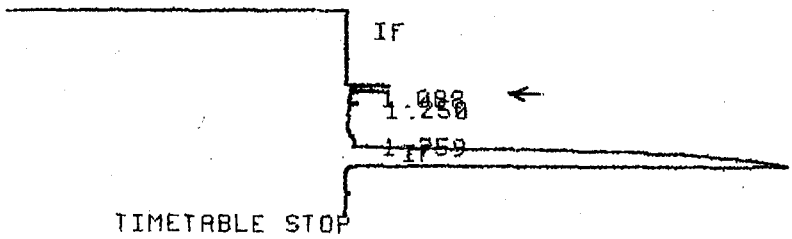
RUN PARAMETERS
ZERO = 20
ATT 2^ = 5
CHT SP = 1.0
AR REJ = 2000
THRSH = 3
PK WD = 0.06

RUN # 1221 AUG 29, 1995 11:15:13
START



Storing processed peaks to A:Q1439DC2.PRO

RUN# 1221 AUG 29, 1995 11:15:13

SAMPLE NAME: CONTROL#2 SAMPLE# 3
FINAL VOL 1ML/5G ALIQUOT

PEAK FILE : A:Q1439DC2.PRO

B95/34-39 (PECANS) 2UL INJ

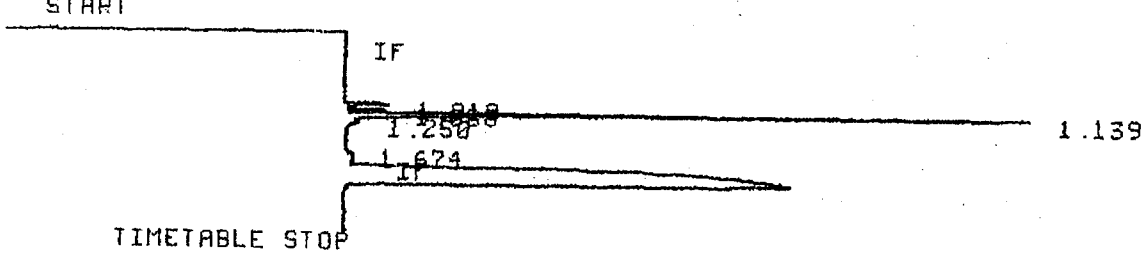
HEIGHT%

RT	HEIGHT	TYPE	WIDTH	HEIGHT%
1.088	10119	PB	.017	66.63373
1.250	2913	BV	.031	19.18214
1.759	2154	PV	.162	14.18412

TOTAL HEIGHT= 15186
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 20
ATT 2^ = 5
CHT SP = 1.0
AR REJ = 2000
THRSH = 3
PK WD = 0.06

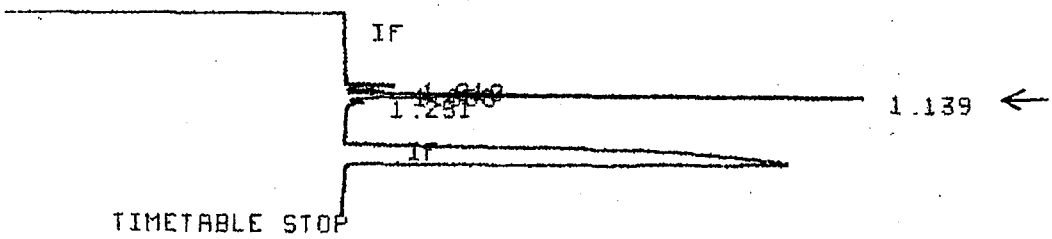
RUN # 1222 AUG 29, 1995 11:20:24
START



28

RUN PARAMETERS
ZERO = 20
ATT 2^ = 5
CHT SP = 1.0
AR REJ = 2000
THRSH = 3
PK WD = 0.06

RUN # 1232 AUG 29, 1995 12:34:18
START



Storing processed peaks to A:Q143B04B.PRO

RUN# 1232 AUG 29, 1995 12:34:18

SAMPLE NAME: 0.05PPM SPK#/ SAMPLE# 5
FINAL VOL 1ML/5G ALIQUOT

PEAK FILE : A:Q143B04B.PRO

B95/34-39 (PECANS) 2UL INJ

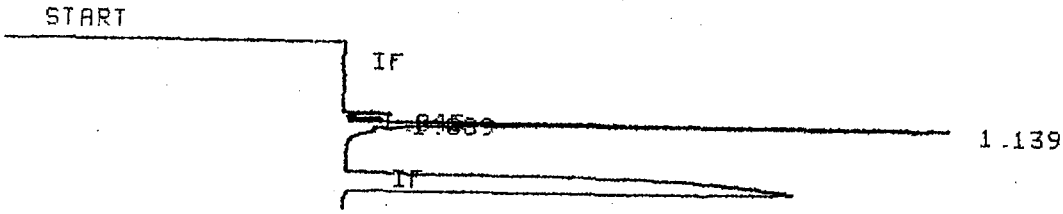
HEIGHT%

RT	HEIGHT	TYPE	WIDTH	HEIGHT%
1.010	12107	PP	.020	7.97368
1.088	9140	PP	.014	6.01961
1.139	127017	PB	.021	83.65354
1.251	3573	PB	.023	2.35318

TOTAL HEIGHT= 151837
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 20
ATT 2^ = 5
CHT SP = 1.0
AR REJ = 2000
THRSH = 3
PK WD = 0.06

RUN # 1233 AUG 29, 1995 12:39:39
START



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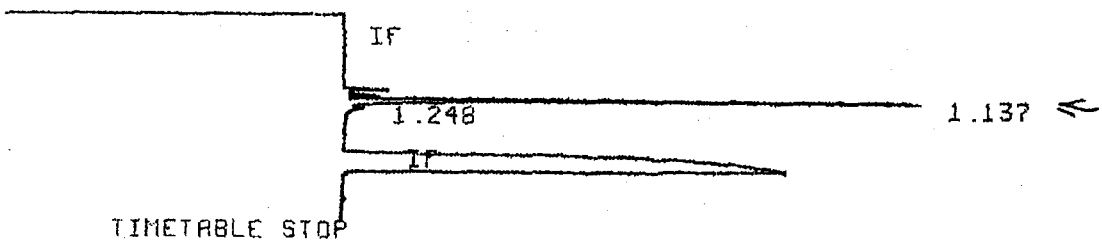
RUN PARAMETERS
ZERO = 20
ATT 2^ = 5
CHT SP = 1.0
AR REJ = 2000
THRSH = 3
PK WD = 0.06

RUN # 1239 AUG 29, 1995 13:11:46
START



Storing processed peaks to A:Q143B913.PRO

RUN# 1239 AUG 29, 1995 13:11:46

SAMPLE NAME: 0.1PPM SPK#1 SAMPLE# 8
FINAL VOL 2ML/5G ALIQUOT

PEAK FILE : A:Q143B913.PRO

B95/34-39 (PECANS) 2UL INJ

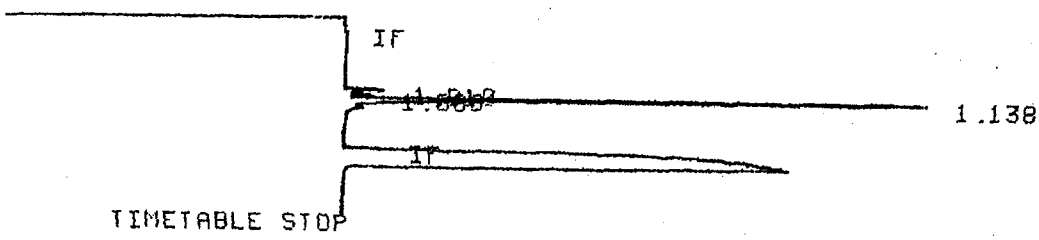
HEIGHT%

RT	HEIGHT	TYPE	WIDTH	HEIGHT%
1.137	136306	PB	.022	97.70829
1.248	3197	BB	.013	2.29171

TOTAL HEIGHT= 139503
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 20
ATT 2^ = 5
CHT SP = 1.0
AR REJ = 2000
THRSH = 3
PK WD = 0.06

RUN # 1240 AUG 29, 1995 13:17:06
START



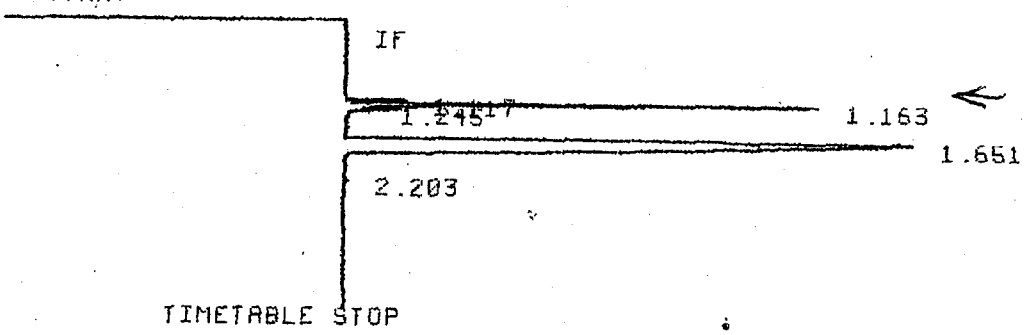
30

1.165	110459	PB	.020	43.91850
1.653	124705	PB	.048	49.58272
2.207	1251	PP	.034	.49740

TOTAL HEIGHT= 251509
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 20
ATT 2^ = 5
CHT SP = 1.0
AR REJ = 2000
THRSH = 3
PK WD = 0.04

RUN # 1075 AUG 19, 1995 13:40:50
START



Storing processed peaks to A:Q13690E3.PRO

RUN# 1075 AUG 19, 1995 13:40:50

SAMPLE NAME: 0.25UG/ML SAMPLE# 7
DMS STD

PEAK FILE : A:Q13690E3.PRO

B95/34-39 (ORANGES) 2UL INJ

HEIGHT%

	RT	HEIGHT	TYPE	WIDTH	HEIGHT%
	1.117	15068	PV	.017	6.15988
TMS	1.163	118768	VV	.021	48.55302
	1.245	13158	VB	.021	5.37907
	1.651	96244	PB	.027	39.34509
	2.203	1377	PP	.052	.56293

TOTAL HEIGHT= 244615
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 20
ATT 2^ = 5
CHT SP = 1.0

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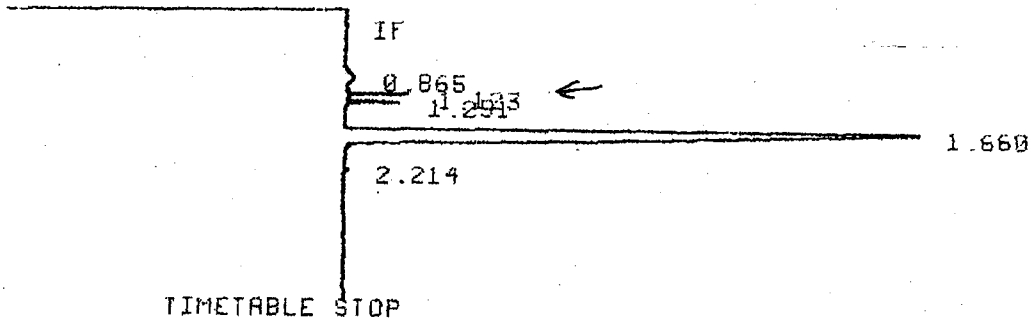
RUN # 1064 AUG 19, 1995 12:36:40
START



Storing processed peaks to A:Q1368109.PRO

RUN# 1064 AUG 19, 1995 12:36:40

SAMPLE NAME: CONTROL#1 SAMPLE# 2
FINAL VOL1ML/5G ALIQUOT

PEAK FILE : A:Q1368109.PRO

B95/34-39 (ORANGES) 2UL INJ

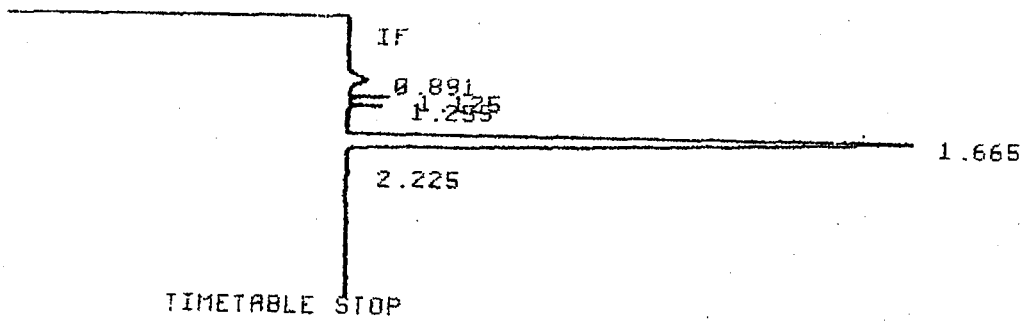
HEIGHT%

RT	HEIGHT	TYPE	WIDTH	HEIGHT%
.865	2353	BV	.142	1.65205
1.123	16199	VV	.016	11.37339
1.251	13339	VV	.021	9.36537
1.660	109064	PB	.052	76.57430
2.214	1474	VP	.039	1.03490

TOTAL HEIGHT= 142429
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 20
ATT 2^ = 5
CHT SP = 1.0
AR REJ = 2000
THRSH = 3
PK WD = 0.04

RUN # 1065 AUG 19, 1995 12:42:32
START

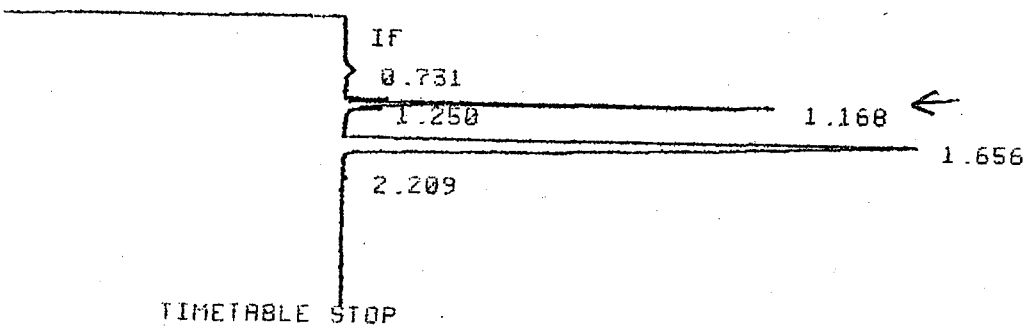


32

TOTAL HEIGHT= 109984
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 20
ATT 2^ = 5
CHT SP = 1.0
AR REJ = 2000
THRSH = 3
PK WD = 0.04

RUN # 1071 AUG 19, 1995 13:17:02
START



Storing processed peaks to A:Q1368B4F.PRO

RUN# 1071 AUG 19, 1995 13:17:02

SAMPLE NAME: 0.05PPM SPK# SAMPLE# 5
FINAL VOL 1ML/5G ALIQUOT

PEAK FILE : A:Q1368B4F.PRO

895/34-39 (ORANGES) 2UL INJ

HEIGHT%

RT	HEIGHT	TYPE	WIDTH	HEIGHT%
.731	2239	VP	.135	.91650
1.168	103408	BV	.021	42.32862
1.250	8494	VB	.021	3.47690
1.656	128886	BB	.056	52.75770
2.209	1271	BV	.045	.52027

TOTAL HEIGHT= 244298
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 20
ATT 2^ = 5
CHT SP = 1.0
AR REJ = 2000
THRSH = 3
PK WD = 0.04

33

TOTAL HEIGHT= 22[illegible]
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 20
ATT 2^ = 5
CHT SP = 1.0
AR REJ = 2000
THRSH = 3
PK WD = 0.04

RUN # 1091 AUG 19, 1995 14:57:23
START

IF
0.537
1.112 1.156
TIMETABLE STOP 1.645

Storing processed peaks to A:Q136A204.PRO

RUN# 1091 AUG 19, 1995 14:57:23

SAMPLE NAME: 0.5PPM SPK#1 SAMPLE# 8
FINAL VOL 10ML/5G ALIQUOT

PEAK FILE : A:Q136A204.PRO

B95/34-39 (ORANGES) 2UL INJ

HEIGHT%

RT	HEIGHT	TYPE	WIDTH	HEIGHT%
.537	1896	BP	.110	.79938
1.112	13316	PU	.017	5.61418
1.156	75239	VV	.023	31.72165
1.239	11545	VV	.023	4.86751
1.645	135189	BP	.122	56.99728

TOTAL HEIGHT= 237185
MUL FACTOR=1.0000E+00

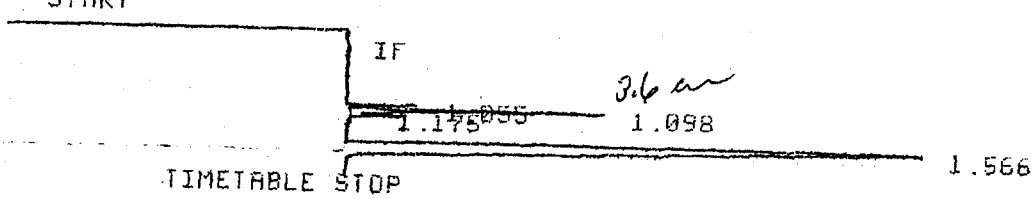
RUN PARAMETERS
ZERO = 20
ATT 2^ = 5
CHT SP = 1.0
AR REJ = 2000
THRSH = 3
PK WD = 0.04

RUN # 1092 AUG 19, 1995 15:01:23
START

IF
0.460

34

```
NO RUN PEAKS STORED


RUN PARAMETERS
ZERO    =  20
ATT 2^  =  5
CHT SP  =  1.0
AR REJ  =  2000
THRSH   =  3
PK WD   =  0.04


*  EDIT  SEQ

1 = ALS INFORMATION
2 = EQUILIBRATION TIME DELAY
3 = METHOD FILE SPECIFICATION
4 = SAMPLE INFORMATION TABLE

SECTION TO BE EDITED: 1

ALS INFORMATION
INET SAMPLER CONTROL [Y*/N]:


7673A SAMPLER:
 LOOP ADDRESS:  8

 FRONT INJECTOR
  INJ/BOTTLE          2 -->     @
  FIRST BOTTLE        7 -->    12  @
  LAST BOTTLE        13 -->      BREAK

*  SEQ  START
RUN # 1099      AUG 19, 1995  15:27:39
START
```




```
Storing processed peaks to A:Q136A9EC.PRO

RUN#  1099            AUG 19, 1995  15:27:39

SAMPLE NAME: 1PPM SPK#2        SAMPLE#   12
FINAL VOL 20ML/5G ALIQUOT

PEAK FILE  : A:Q136A9EC.PRO

B95/34-39 (ORANGES) 2UL INJ

HEIGHT%
```

RT	HEIGHT	TYPE	WIDTH	HEIGHT%
1.055	16919	PU	.016	7.69164
1.098	62111	VV	.023	28.23663
1.175	14077	VB	.019	6.39962
1.566	126859	PB	.056	57.67210

TOTAL HEIGHT [illegible]

35